



Technical Bulletin

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Radleys Contact

techsupport@radleys.co.uk

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Reactor-Ready vacuum and pressure testing

This document describes Radleys' recommended protocols for leak testing Reactor-Ready jacketed lab reactors to check gas tightness. Similar methods can be used for testing other reaction systems.

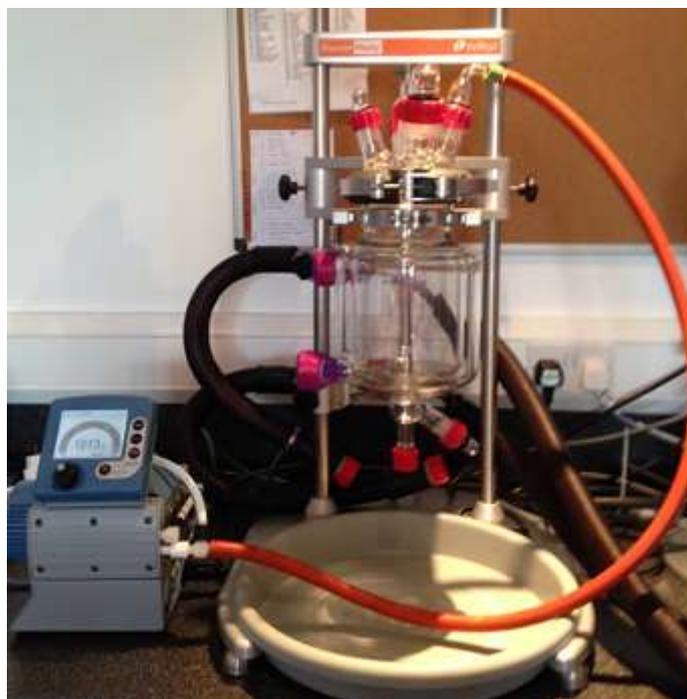
Vacuum testing

Parts required

- Reactor-Ready – including vessel, lid, PTFE support collar and O-ring, piston, stirrer guide, stirrer shaft, Pt100 and Pt100 adapter
- Suitable vacuum pump; for example, Vacuubrand MD1C Vario pump with CVC controller
- Suitable vacuum tubing; we use 18 mm OD x 8 mm ID
- Glass cone stoppers of appropriate sizes; the Radleys part numbers are:
 - 2 x RR139027 Glass Cone Stopper B19 (for lid port and bottom outlet valve (BOV) run-off)
 - 2 x RR139029 Glass Cone Stopper B24 (for lid ports)
 - 1 x RR139031 Glass Cone Stopper B29 (for lid port)
 - 1 x RR139033 Glass Cone Stopper B34 (for lid port)

N.B. You will not need a glass cone stopper for the lid port you will be inserting the adapter for vacuum tubing into

- Right angle adapter (or gas purge adapter) to fit into a lid port to connect to vacuum tubing; we use: RR139011 Right Angle Adapter B29 + GL14 + PTFE fittings
- GL25 cap to seal BOV in place of piston; we use one from RR139117 Screw Cap Closed with PTFE Seal GL25 (Pack of 10)
- Vacuum (silicone) grease



Method

1. Ensure all items are clean, completely dry, and not damaged.
2. Place vessel in Reactor-Ready framework, with collar/O-ring, lid and clamp in place.
3. Insert right angle adapter into port on lid. Connect vacuum tubing between this and the vacuum pump.
4. Insert glass cone stoppers into all remaining lid ports, using vacuum grease on the joints.
5. Do not insert piston into vessel at this stage. Insert glass cone stopper (with vacuum grease) into BOV run-off. In place of piston, add the GL25 screw cap.
6. The set-up should look like the photo shown on the previous page.
7. Start the vacuum pump. Wait for it to reach the best possible vacuum. (Note that when vacuum pumps and vacuum tubing are new, you may need to run the vacuum for some hours before the best vacuum levels can be achieved.) Record the vacuum level.
8. Stop the vacuum pump and carefully release the vacuum. Next, remove the screw cap from the BOV, and insert the piston. Re-connect the vacuum tubing and start the vacuum pump. Wait for the pressure to decrease until the vacuum level plateaus. Record the value.
9. Repeat step 8, but this time add a stirrer guide and stirrer shaft to the lid. (Keep the piston in place.)
10. Repeat step 8, but this time add a Pt100 with appropriate adapter. Make sure the section of the Pt100 secured in the adapter is straight not bent, to ensure a good seal. (Keep the piston and stirrer guide/shaft in place.)
11. Check the leak rate by switching off the vacuum pump and observing how much the vacuum level changes in 1 minute.

Results

- The Reactor-Ready reaction vessels are typically expected to achieve around 10 mbar. However, in previous testing, with all accessories in place as described above, Radleys have achieved approximately 3-5 mbar, with a leak rate of about 5 mbar/min.
- If the introduction of an accessory impairs the vacuum level, examine that item to try to identify why it has caused a leak.
- Please note that any moisture in the system will increase the pressure (and therefore reduce the vacuum level).

Pressure testing

Parts required

- Reactor-Ready – including vessel, lid, PTFE support collar and O-ring, piston, stirrer guide, stirrer shaft, Pt100 and Pt100 adapter
- Suitable manometer (pressure gauge), such as Digitron 2082P Manometer
- Manual pressure pump; we use 2911 Gauge Handpump from DRUCK & TEMPERATUR Leitenberger
- Tubing suitable for slight positive pressure; we recommend nylon 6 mm OD x 4 mm ID part no. NTM06/040B-10 from Hoses Direct
- T piece for connecting tubing to pressure pump, manometer and lid fitting; we use part no. 2019-8404 from Hoses Direct
- Glass cone stoppers of appropriate sizes; the Radleys part numbers are:
 - 2 x RR139027 Glass Cone Stopper B19 (for lid port and bottom outlet valve (BOV) run-off)
 - 2 x RR139029 Glass Cone Stopper B24 (for lid ports)
 - 1 x RR139031 Glass Cone Stopper B29 (for lid port)
 - 1 x RR139033 Glass Cone Stopper B34 (for lid port)

N.B. You will not need a glass cone stopper for the lid port you will be inserting the adapter for tubing into

- Right angle adapter (or gas purge adapter) to fit into a lid port to connect to pressure tubing; we use: RR139011 Right Angle Adapter B29, plus Bohlender GL15 compression fitting (in place of the usual GL14 hose barb)
- GL25 cap to seal BOV in place of piston; we use one from RR139117 Screw Cap Closed with PTFE Seal GL25 (Pack of 10)
- Vacuum (silicone) grease

Method

1. Ensure all items are clean, completely dry and not damaged.
2. Place vessel in Reactor-Ready framework, with collar/O-ring, lid and clamp in place.
3. Insert right angle adapter into port on lid. Connect tubing to this and the T-piece. Connect the T-piece to the manometer (pressure gauge) and the hand pressure pump. This is shown in the photo on the right.
4. Insert glass cone stoppers into all remaining lid ports, using vacuum grease on the joints.
5. Do not insert piston into vessel at this stage. Insert glass cone stopper (with vacuum grease) into BOV run-off. In place of piston, add the GL25 screw cap.
6. Pump the hand pump until the pressure is at 500 mbar (i.e. 0.5 barg), our recommended maximum pressure for glassware. Wait to see whether the pressure holds. It may drop a few mbar initially, but it should then stabilise.
7. Carefully release the pressure (via pressure relief valve on hand pump). Next, remove the screw cap from the BOV, and insert the piston. Repeat step 6.
8. Repeat step 7, but this time add a stirrer guide and stirrer shaft to the lid. (Keep the piston in place.)
9. Repeat step 7, but this time add a Pt100 with appropriate adapter. Make sure the section of the Pt100 secured in the adapter is straight not bent, to ensure a good seal. (Keep the piston and stirrer guide/shaft in place.)



Results

- With all accessories in place as described above, the pressure should be stable at around 500 mbar, with no (obvious) leak rate.
- If the addition of an accessory introduces an air leak, examine that item to try to identify why it has caused a leak.